

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Non-Sweating Lipstick Formulation

We, THE MEARL CORPORATION, a corporation of the State of New Jersey, United States of America, having a principal place of business at 217 North Highland Avenue, Ossining, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

In the following specification all parts and percentages are given by weight, unless otherwise indicated.

A serious problem in lipstick formulations, particularly in connection with relatively soft lipsticks, is the phenomenon known as "sweating". Sweating of a lipstick occurs when droplets of oil migrate to the surface of the stick during storage at relatively high temperatures. While such droplets may be re-absorbed into the stick as, for example, when the storage temperature falls, the appearance of the surface of the lipstick nevertheless remains marred.

Lipsticks containing nacreous pigments, which impart a pearly luster to the lips, generally have appreciable contents of low viscosity oils to insure good application and orientation of the nacreous pigment platelets. Such lipsticks thus comprise rather soft formulations and are, therefore, particularly subject to the sweating phenomenon. Accordingly, it has been necessary to limit storage conditions of heretofore proposed nacreous lipstick formulations to a relatively restricted range of temperatures.

It is among the objects of the present invention to provide a nacreous pigment paste for a non-sweating lipstick formulation, and such a formulation, which latter is substantially free from "sweating", as defined herein.

A further object of the invention is to provide such a formulation which may be stored under a broader range of temperature conditions than previously available nacreous lipsticks, without any adverse effects resulting from the sweating phenomenon.

Other objects and advantages of the invention will be apparent from the following detailed description of preferred forms thereof.

It has been discovered, in accordance with the present invention, that the sweating of nacreous lipstick formulations may be retarded, if not completely prevented, by incorporating therein nacreous pigment pastes including water-insoluble cellulose ester or cellulose ether sweating inhibitors. The cellulose compounds so effective include ethyl cellulose, nitrocellulose, cellulose acetate butyrate, and cellulose acetate propionate. Such materials, when incorporated in nacreous lipstick formulations in amounts of from 0.1% to 2% by weight thereof, have been found to effectively inhibit sweating of such formulations.

It has previously been proposed to incorporate cellulose derivatives in cosmetic preparations as film-formers. Thus, the use of ethyl cellulose as a film former in a liquid rouge preparation has been suggested in Klimist U.S. patent No. 2,230,063 of January 28, 1941. Similarly, Wotzilka *et al* U.S. patent No. 3,122,481 of February 25, 1964 suggests the application of a water-soluble cellulose ether, viz., methyl cellulose, as a film-forming base for a lipstick. It has not, however, previously been proposed to utilize water-insoluble cellulose esters or ethers as sweating inhibitors in nacreous pigment pastes for use in lipstick formulations, or in wax and oil base solid lipsticks including the same.

Desirably, the cellulose compound is initially dissolved in the nacreous pigment paste vehicle, and such paste containing the cellulose derivative is thereafter blended with the lipstick base. However, the cellulose compound may rather be added to the lipstick base either before or after the incorporation of the nacreous pigment therein. It will be understood, therefore, that the reference in the following specification and claims to lipstick formulations containing nacreous pigment pastes, one constituent of which comprises the water-insoluble cellulose compound sweat-inhibitor, is intended to in-

[Price 4s. 6d.]

clude such formulations in which the cellulose compound may have been incorporated either before, after, or concurrently with, the addition of the nacreous pigment thereto.

5 The nacreous pigments which may be utilized in the lipstick formulation of the present invention include natural pearl essence crystals, which consist primarily of guanine; bismuth oxychloride; and mica flakes provided with
10 appropriate coatings, e.g., titanium dioxide. Such pigments contain oriented plate-like particles of high refractive index, and produce a pearly luster on reflection of light from multiple layers of such particles. Those
15 nacreous pigments employed in the lipstick formulation are, of course, non-toxic.

Desirably, the nacreous pigments are utilized in the form of pigment pastes in which the light-reflecting platelets are dispersed in a
20 suitable vehicles. Such vehicles include, for example, castor oil, isopropyl myristate, and oleyl alcohol.

The concentration of the nacreous pigment in the pigment paste may be varied, depending
25 upon the particular pigments and vehicles utilized. Thus, natural pearl essence paste useful in lipstick formulations generally consists of from 20% to 22% pearl essence crystals in castor oil, whereas such pastes incorporating bismuth oxychloride as a nacreous
30 pigment may conveniently have a 70% pigment concentration in such vehicle. The cellulose compound, when incorporated in the nacreous pigment paste, is admixed therein in concentrations of from 0.3% to 5.6%, preferably
35 from 1% to 3%, by weight of the paste. While higher concentrations also inhibit sweating, such are less desirable because they are relatively uneconomical and may change
40 the texture of the finished lipstick.

The nacreous pigment paste may be incorporated in any conventional lipstick base including a wax component which may be cast into the desired lipstick shape, suitable oil and plasticizer components, and the necessary pigment constituents, both colored and white. Such wax, oil and pigment ingredients are disclosed, for example, in Buchwalter et al U.S. patent No. 3,211,619 of October 12, 1965, the applicable disclosure of which is incorporated
45 herein by reference. 50

The lipstick formulation may contain from 5% to 50% of the nacreous pigment paste, the proportion being correlated with the concentration of the cellulose compound in order that the concentration of the latter in the lipstick formulation ranges, as indicated above, from 0.1% to 2%, preferably from 0.36% to 1.1%, by weight of the composition. Lipstick formulations containing 36% by weight of the nacreous pigment paste are frequently commercially employed and are, therefore, referred to in the following examples by way of illustration. It will, however, be understood that the formulations of the present invention may
55 incorporate different concentrations of the nacreous pigment paste sufficient to provide the proportion of the cellulose compound sweating inhibitor referred to hereinabove. Moreover, it is intended that the lipstick formulations given in the following examples are illustrative only, other conventional lipstick formulations being useful in the composition of the present invention as well. 60 65 70

EXAMPLE 1

75 A pearl lipstick was prepared with the following composition, all percentages being by weight:

Nacreous pigment paste	36.5%
Candelilla wax	11.2
Oleyl alcohol	31.7
Isopropyl myristate	6.1
Dispersion of color in castor oil	14.3
2,5-di-t-butyl hydroquinone	0.2
	<hr/> 100%

The nacreous pigment paste comprised:

Natural pearl essence crystals	22.0%
Nitrocellulose, 1/2 second (RS type, manufactured by the Hercules Powder Co., which is soluble in esters and other solvents)	2.0
Castor oil	76.0
	<hr/> 100%

5 All the components except the nacreous pigment were mixed in a master batch, which was then blended with the nacreous pigment with slow stirring at 85° C for about 20 minutes. After air was permitted to escape, the lipsticks were cast in molds which were chilled. Sweating was evaluated by placing the lipstick samples in closed metal lipstick cases in an oven at 40° C. No sweat droplets appeared in 96 hours.

For comparison, the identical formulation, save for the omission of the nitrocellulose inhibitor, was similarly tested. After 2 hours, considerable sweating, rated on an arbitrary appearance scale as 3+, was observed. 15

EXAMPLE 2

The procedure of Example 1 was repeated except that the nacreous pigment paste comprised: 20

Bismuth oxychloride crystals	70.0%
Ethyl cellulose N-300 (a product of the Hercules Powder Co. having an ethoxyl content of 47.5—49.0% and a viscosity of 250—350 cps when measured at 25°C. in a 5% solution in 80:20 toluene: ethanol by weight)	2.0
Castor oil	28.0
	<hr/> 100.0%

25 Employing the same sweating test as in Example 1, no sweating occurred after storage at 96 hours at 40° C., followed by 48 hours at 41° C. Sweating, rated at 1+ on the same arbitrary scale as employed in Example 1, occurred after an additional 2 hours at 42.0° C. For comparison, the same composition,

minus the ethyl cellulose, exhibited sweating rated at 1+ after 2 hours storage at 40.0° C. 30

EXAMPLE 3

The procedure of Example 1 was again repeated, except that the nacreous pigment paste comprised:

Titanium dioxide-coated mica (MEARLIN*-AC, a product of the Mearl Corporation)	40.0%
Ethyl cellulose N—300	2.0
Castor Oil	58.0
	<hr/> 100.0%

* MEARLIN is a Trade Mark

No sweating was encountered until the temperature was raised to 43° C. in the following stages:

- 5 96 hours at 40° C.
 48 hours at 41° C.
 24 hours at 42° C.
 4 hours at 43° C.

EXAMPLES 4—8

The nacreous pigment paste of Example 1 was replaced, in the lipsticks of the following Examples, by 70% bismuth oxychloride pastes in castor oil, containing the following cellulose derivatives:

10

TABLE I

Similar Lipstick Formulations Containing Varying Cellulose Compound Sweating Inhibitors

Example	Cellulose Compound	Concentration	Sweating Behavior	
			at 40°C.	at 41.0°C. ¹
4	Ethyl Cellulose K—100 ²	2%	0 (24 hrs.)	0.80+
5	Ethyl Cellulose K—200 ³	2%	0 (24 hrs.)	0.85+
6	Ethyl Cellulose N—200 ⁴	2%	0 (24 hrs.)	0.00+
7	Cellulose Acetate Butyrate EAB—500—1 ⁵	2%	0 (24 hrs.)	1.00+
8	Cellulose Acetate Butyrate EAB—500—5 ⁶	2%	0 (24 hrs.)	1.05+
Control	None	None	1+(2 hrs.)	Failed Badly

¹ After initial storage at 40.0°C. for 24 hours, followed by heating at 41.0°C. for 2 hours.

² A product of the Hercules Powder Co. having an ethoxyl content of 45.5—46.8% and a viscosity of 80—105 cps in a 5% solution in 80:20 toluene: ethanol by weight.

³ A product of the Hercules Powder Co. having an ethoxyl content of 45.5—46.8% and a viscosity of 150—250 cps in a 5% solution in 80:20 toluene-ethanol by weight.

⁴ A product of the Hercules Powder Co. having an ethoxyl content of 47.5—49.0% and a viscosity of 150—250 cps in a 5% solution in 80:20 toluene: ethanol by weight.

⁵ A product of the Eastman Chemical Products, Inc. having a butyryl content of 50% and a viscosity of 3.0—4.5 poises in a solution containing 20% cellulose acetate butyrate, 72% acetone, and 8% ethanol

⁶ A product of the Eastman Chemical Products, Inc. having a butyryl content of 50% and a viscosity of 15.0—22.5 poises in a solution containing 20% cellulose acetate butyrate, 72% acetone, and 8% ethanol.

EXAMPLES 9—12
 Example 1 was repeated, replacing nitro-cellulose with various concentrations of ethyl cellulose N—300; the effect of concentration variation is indicated in the following Table, for Example 9—12: 5

TABLE II

Similar Lipstick Formulations Containing Varying Concentrations of the Same Cellulose Compound Sweating Inhibitor

Example	Ethyl Cellulose Concentration	Sweating Behavior at 40°C.
9	0.5%	2+ (2 hrs.)
10	1.0	1+ (2 hrs.)
11	3.0	0 (120 hrs.)
12	4.0	0 (120 hrs.)
Control	0.0	3+ (2 hrs.)

EXAMPLE 13
 A nacreous pigment paste constituted of 10 70.0% bismuth oxychloride, 2.0% ethyl cellulose N—200 and 28.0% oleyl alcohol was incorporated in the following lipstick formulation:

Nacreous pigment	36.5%
Candelilla wax	11.2
Oleyl alcohol	21.4
Isopropyl myristate	6.1
Color in castor oil	14.3
2,5-di-t-butyl hydroquinone	0.2
Castor Oil	10.3
	<u>100.0%</u>

15 No sweating was obtained after the following heat history: appearing on the bottom of Table I above), exhibited 1+ sweating after 2 hours storage at 40.0° C.

20 24 hours at 40.0° C
 24 hours at 41.0° C
 24 hours at 42.0° C
 24 hours at 43.0° C
 72 hours at 44.0° C

25 For comparison, it will be noted that a formulation containing an equivalent amount of bismuth oxychloride (in castor oil), without the ethyl cellulose additive (see the control

EXAMPLE 14
 A lipstick formulation having a composition 30 similar to that prepared in Example 2 was formed by adding the nacreous pigment paste, to which the cellulose derivative had not been added, to the lipstick base containing the indicated amount of ethyl cellulose. The resulting 35 formulation contained the following ingredients:

Nacreous pigment paste	36.50%
Candelilla wax	11.20
Oleyl alcohol	30.97
Ethyl cellulose N—300	0.73
Isopropyl myristate	6.10
Color in castor oil	14.30
2,5-di- <i>t</i> -butyl hydroquinone	0.20
	<hr/> 100%

The same sweating behavior was observed as in Example 2.

- 5 It is clear from the above examples that the incorporation of the specified water-insoluble cellulose compounds in nacreous lipstick formulations results in markedly decreased sweating characteristics thereof. Such lipsticks retain a desirable soft consistency, permitting easy application, and yet may be stored at higher temperatures than usual without concern. While preferred embodiments of the composition of the present invention have been illustrated above, it is to be understood that the invention is not limited to these precise compositions and that changes may be made therein without departing from the scope of the invention. The preceding description should, therefore, be construed as illustrative and not in a limiting sense.

WHAT WE CLAIM IS:—

1. A nacreous pigment paste for a non-sweating (as hereinbefore defined) lipstick formulation, comprising a dispersion of a nacreous pigment in a vehicle therefore, said dispersion containing from 0.3% to 5.6% by weight of a water-insoluble cellulose ester or ether.
2. The nacreous pigment paste of claim 1, containing from 20% to 70% by weight of said nacreous pigment.
3. The nacreous pigment paste of claim 1, in which said water-insoluble cellulose compound is dissolved in said vehicle and is ethyl cellulose, nitrocellulose, cellulose acetate butyrate, or cellulose acetate propionate.
4. The nacreous pigment paste of claim 1, in which the nacreous pigment is constituted of natural pearl essence, bismuth oxychloride, or coated mica flakes.
5. The nacreous pigment paste of claim 1,

wherein the nacreous pigment is dispersed in a castor oil vehicle, and the water-insoluble cellulose compound admixed therewith is ethyl cellulose.

6. A non-sweating lipstick formulation, comprising a solid wax base having uniformly dispersed therethrough a colored pigment and the nacreous pigment paste of claim 1, the lipstick containing from 5% to 50% by weight of the nacreous pigment paste and from 0.1% to 2% by weight of said water-insoluble cellulose compound.

7. The non-sweating lipstick formulation of Claim 6, in which said water-insoluble cellulose compound is dissolved in said vehicle and is ethyl cellulose, nitrocellulose, cellulose acetate butyrate or cellulose acetate propionate.

8. The non-sweating lipstick formulation of claim 6, in which the nacreous pigment is constituted of natural pearl essence, bismuth oxychloride, or coated mica flakes.

9. The non-sweating lipstick formulation of claim 6, in which said water-insoluble cellulose compound is present in an amount of from 0.36% to 1.1% by weight thereof.

10. The non-sweating lipstick formulation of claim 6, wherein the nacreous pigment is dispersed in a castor oil vehicle and the water-insoluble cellulose compound admixed therewith is ethyl cellulose.

11. A nacreous pigment paste as claimed in claim 1 substantially as hereinbefore described.

12. A non-sweating lipstick formulation as claimed in claim 6 substantially as hereinbefore described.

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